

EXHIBIT D9

ITALIAN, MEDICATED, GRANTHAM TALC

From R. Rello's files

EXHIBIT

tabbies

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RESEARCH
AND
DEVELOPMENT

WALTER C. McCRONE ASSOCIATES, INC.

- SMALL PARTICLE PROBLEMS
- SOLID STATE CHEMISTRY
- ULTRAMICRO ANALYSIS
- CRYSTALLOGRAPHY
- MICROSCOPY

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3 September 1971

Dr. A. Goudie
Johnson and Johnson
Research Center
501 George Street
New Brunswick, New Jersey 08901

Dear Dr. Goudie:

Enclosed are two copies of our preliminary report on the Grantham ore and the Shower to Shower and medicated powders. We have other photographs of the Grantham ore if you find that you need more of any particular type. Please let us know what type you wish and we will send them to you. There are three sets of photographs, and I will send a fourth set with a copy of our final report.

I am sorry that it took the extra time, but our microscope was down for over two weeks while we got service on it. We were also trying to refine our techniques in selected area electron diffraction similar to Dr. Pooley's technique. I think we have done rather well, and I believe that our detection limits are as low as possible. The fiber of chrysotile in the G-11 sample was about 200A by 2000A, and the pattern was very clear. If it is necessary, we can probably go to fibers 200A by 500-1000A long.

I believe this gives us an edge in future discussions with Langer or anyone else. I do not believe he knows what he is talking about. These fibers do not decompose in the beam, and you can identify them clearly with selected area electron diffraction. I think it is possibly the only way. The tube characteristic is not always very clear in chrysotile, but you generally have some indication. With tremolite, you generally have lines running parallel to the axis and not the mottled appearance of rolled talc with subgrain boundaries.

I marvel at Dr. Pooley's ability to look at photographs and decide what they were. Two months later, I am beginning to do that now. The little characteristics of each fiber are beginning to fit into place.

Dr. A. Goudie

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I have just re-examined the G-11 sample and find no chrysotile in the second sample. There was one fiber, but it gave a different diffraction pattern from that of chrysotile.

Well, I am now headed for northern Michigan for a week. When I get back, we can exchange stories on the big one that got away.

Good luck.

Very truly yours,

Gene R. Grieger

Gene R. Grieger
Research Physicist

GRG:rlt
Enclosures
Ref: MA2347

Report to:

Johnson and Johnson
Research Center
501 George Street
New Brunswick, New Jersey 08901

Project No. MA-2347

PRELIMINARY REPORT ON EXAMINATION OF GRANTHAM ORE, MEDICATED
TALCUM POWDER AND SHOWER TO SHOWER TALCUM POWDER

Submitted by:

Walter C. McCrone Associates, Inc.
493 East 31st Street
Chicago, Illinois 60616

No. of Copies

3 September 1971

Walter C. McCrone Associates, Inc.

Project No. MA-2347

PRELIMINARY REPORT ON EXAMINATION OF GRANTHAM ORE, MEDICATED
TALCUM POWDER AND SHOWER TO SHOWER TALCUM POWDER

An examination has been made of the following samples submitted by Johnson
and Johnson Research Center:

- (1) Grantham ore,
- (2) Grantham ore composite,
- (3) Vermont ore (Raymond mill, unconditioned),
- (4) Johnson's medicated talcum powder, and
- (5) Johnson's Shower to Shower talcum powder.

The results of this examination are summarized below.

The Grantham ore was found to contain about 0.01-0.1% tremolite; this is reduced by a factor of between 2-5 by beneficiation. The ore is also believed to contain chrysotile in the range of 0.001-0.01%, reduced by a factor of between 5-10 by beneficiation. Both the tremolite and the chrysotile were in sufficient quantities to be identified by electron diffraction. There are also much larger quantities of rolled talc in this ore than in the previously examined samples of Vermont ore, although this too is reduced by beneficiation. There are large quantities of extraneous materials such as calcite and chlorite, some of which decompose in the beam into a gas phase and a fine deposit. Other minerals present decompose into a spherical ball, and some of these have been tentatively identified by electron diffraction as follows:

<u>Compound</u>	<u>ASTM Card</u>	<u>Relative Abundance</u>
Na_2PbO_3	8-245	rare
Na_3AlF_6	1-1273	common
KClO_2	2-1320	medium

Electron microprobe studies showed some asbestos present, some potassium feldspar with inclusions of pyrites and iron oxides, considerable amounts of calcite and traces of graphite.

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In the sample of Vermont ore (Raymond mill, unconditioned), we found no asbestos; however, there was more rolled talc and extraneous minerals than in the Vermont ore previously studied (MA-2330-2).

In the medicated powder, we found one fiber of chrysotile, and we estimate that this powder contains less than 0.001% asbestos; the correct order of magnitude is probably 0.0001% since much of the sample remains at the bottom of the test tube. The tricalcium phosphate added as a buffer to this powder does form clusters of needle-like crystals which in some cases have the tube appearance of chrysotile. The electron diffraction pattern, however, is not similar to that of chrysotile.

In the Shower to Shower sample we found several fibers which do not show the coring typical of chrysotile. These may be fine fibers of talc or of $\text{Ca}(\text{PO}_4)_2 \cdot n\text{H}_2\text{O}$ which also occur as needles. There was one very small fiber which could have been chrysotile in a field of fine talc flakes. We were unable to obtain a diffraction pattern from the sample, but we feel strongly that it may be chrysotile. Again, the percentage of chrysotile is very low, in the range of 0.001-0.0001%.

In running the standards of the additives to the medicated powder and to the Shower to Shower powder, we did find two fibers of chrysotile, positively identified both by morphology and diffraction patterns in the G-11 sample. These two fibers were very close together and may have come from the same source. Again, the percentage was low, 0.005-0.001%, but this may be a potential source of contamination of the end product. The tricalcium phosphate and the Microcele showed no contamination by asbestiform minerals.

Our final report on this project will follow shortly. In the meantime, we are enclosing the photographs relevant to this work.

FINAL REPORT ON SHOWER TO SHOWER, MEDICATED POWDER
AND FEMININE SPRAY

Summary

We have examined samples of three Johnson & Johnson talc products, Shower to Shower, Medicated Powder and Feminine Aerosol Spray. These products all seem to be quite clean, and we did not find any fibers of the asbestiform minerals in any of them: in particular, there are no bundles of chrysotile fibers and only in rare cases did we find any fibers suspect on the basis of morphology. The maximum number of suspect fibers which we found in any sample was two, in the Shower to Shower. There is a possibility of contamination by approximately one fiber in our lab and probably some contamination in the processing just from airborne fibers which get into the product during the grinding and mixing procedures.

Walter C. McCrone Associates, Inc.

Introduction

McCrone Associates has examined the appearance and fiber content of samples of Johnson & Johnson Shower to Shower, Medicated Talc Powder and Feminine Aerosol Spray. The electron microscopy and electron diffraction methods used are described in a previous report to Johnson & Johnson, MA2330-1.

Final Results of Examination

Feminine Spray

The Feminine Spray sample showed talc plate sizes which are considerably smaller than those particle sizes in the other talc products.

The fiber content in this sample of Feminine Spray seems to be quite low, and we found very few examples of rolled talc or other fibers. We found one unknown mineral fiber which we considered at first to be chrysotile, but, after a careful comparison of our standard chrysotile electron diffraction patterns and the unknown mineral pattern, we decided that it was not chrysotile. This fiber has a central coring similar to chrysotile but shows a diffraction pattern different from chrysotile. This strong morphological similarity might have led to confusion without the use of electron diffraction and chrysotile standards.

A few talc fibers and some other small mineral fibers were identified with electron diffraction. In addition, we found a fiber from which we could not get an electron diffraction pattern. It did not appear to have a chrysotile structure but might possibly be calcium phosphate or sodium sesquicarbonate.

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Medicated Powder

The Medicated Powder is very similar in cleanliness to the Feminine Spray Powder. The additive "Microcell" appears to be a fine web-like structure, in some cases platy which appears very similar to crinkled newspaper when viewed from the top.

None of the several chemical additives which we found appeared to be fibrous: several orthorhombic, cubic, or perhaps tetragonal crystals might be calcium silicate.

We found few examples of fibrous talc and only one fiber which could be considered suspect because of its central coring. This suspect fiber gave an excellent electron diffraction pattern which, in our opinion, differs from chrysotile both in d values and intensity relationships. It could be hydromagnesite, halloysite or a rolled silicate. In addition to the suspect fiber we found examples of what appeared to be sodium sesquistrate or a chemical very similar to it: the coring was not quite as obvious and it did give a slight electron diffraction pattern; there is a strong possibility that this is calcium phosphate or one of the ingredients added to the powder to give its medicated quality.

Shower to Shower

The fiber content of Shower to Shower is quite low in comparison to previous samples which we have investigated and is predominantly rolled talc, I estimate it to be on the order of 0.001% and definitely no greater than 0.005%. We found three suspect fibers. Of these, two were found in one field and probably have the same source, very possibly contamination. Because they were covered by a talc plate, it was impossible to get electron diffraction

patterns from these two fibers; they appeared to be the same size and to have the same general characteristics, but it still is questionable whether they are chrysotile. We have, however, found traces of chrysotile in G-11, one of the additives to Shower to Shower, and this might be a possible source of these contaminant fibers. The third fiber is quite small and in a field surrounded by small plates of talc. We have compared its electron diffraction pattern with those of talc and chrysotile and it does not appear to be either of the two. It is similar to chrysotile only insofar as it has an apparent coring of the sample.

In the remainder of the sample, we found few talc shards, i.e., small fragments of talc broken from larger plates which give talc patterns in most cases but which appear as fibers. There is some rolled talc and some other mineral fibers which we have not been able to identify with electron diffraction but which do not have any of the characteristics of amphiboles or chrysotile. As in the other two Johnson & Johnson products, we found several examples of fibers which have all of the general characteristics of sodium sesquicarbonate, although they do not produce electron diffraction patterns. They generally occur in bundles and are not limited to single fibers as is chrysotile. The bundles of fibers, however, are quite random in orientation and seem to be almost a woven or intertwined structure.

Observation

In regard to the suspect fibers, many types of mineral fibers have a core similar to chrysotile, but their electron diffraction patterns identify them as other minerals. However, we cannot always obtain electron diffraction patterns from suspect fibers because they may have slightly different mineralogical structures which do not lead to strong electron diffraction patterns and/or their pattern may be masked by the major mineral species present in the sample.